

APPLICATION FOR UNITED STATES PATENT

LUBE HYDROISOMERIZATION SYSTEM

Applicants: **Zhaozhong Jiang**
25 MacIntosh Road
Somerville, New Jersey 08876
Citizenship: U.S.A.

Randall D. Partridge
33 Salters Farm Road
Califon, New Jersey 07830
Citizenship: U.S.A.

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CROSS-REFERENCE TO RELATED APPLICATIONS

This patent application claims benefit of United States Provisional Patent Applications Serial No. 60/416,949 filed October 8, 2002.

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CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims benefit of United States Provisional Patent Application Serial No. 60/416,949 filed October 8, 2002.

FIELD OF THE INVENTION

[0002] The present invention relates to a process for converting waxy feeds to lube basestocks with a reduced viscosity.

BACKGROUND OF THE INVENTION

[0003] There is significant economic incentive to convert wax to high quality lube basestocks, especially base oils with properties and performance comparable to, or better than, those of polyalphaolefins (PAO). The upgrading of wax greatly relies on advanced wax isomerization technology that selectively transforms linear paraffins to multi-branched isoparaffins.

[0004] Processes for converting wax to paraffinic lube basestocks are known. A typical process is a two-stage process that hydroisomerizes wax to a waxy isoparaffins mixture in the first step, followed by either solvent dewaxing or catalytic dewaxing the waxy isoparaffins mixture in the second step to remove residual wax and achieve a target lube pour point.

[0005] The hydroisomerization catalysts disclosed previously, such as Pt supported on amorphous aluminosilicate or Zeolite Beta (Beta), normally

possess large pores that allow the formation of branch structures during paraffin isomerization. Examples of other large pore molecular sieves include ZSM-3, ZSM-12, ZSM-20, MCM-37, MCM-68, ECR-5, SAPO-5, SAPO-37 and USY. However, these large pore catalysts are not selective enough to preferentially convert normal and lightly branched paraffin waxes in the presence of multi-branched isoparaffin molecules. As a result, the isoparaffin products derived from wax often contain residual wax that needs to be dewaxed in order to meet target lube cloud points or pour points. The cloud point of a lube is the temperature at which the first trace of wax starts to separate, causing the lube to become turbid or cloudy (e.g., ASTM D2500). The pour point of a lube is the temperature at which lube and wax crystallize together as a whole and will not flow when poured (e.g., ASTM D97). Dewaxing can be achieved by additionally using either a solvent dewaxing process or a catalytic dewaxing process.

[0006] Most selective dewaxing catalysts used in a catalytic dewaxing process have relatively small pore structures and catalyze lube pour point reduction by selectively cracking normal and lightly branched paraffin waxes. Such dewaxing catalysts usually have low paraffin isomerization selectivity.

[0007] Few catalysts have been reported to be efficient in catalyzing both hydroisomerization and dewaxing of paraffin wax to low pour point lubes. Also, such catalysts have difficulty to convert feeds with a high molecular weight component, as a result of which the lube products often appear hazy (or cloudy).

[0008] There remains a need therefore to achieve conversion of high molecular weight wax and a low enough pour point without sacrificing lube isomerization dewaxing selectivity.

SUMMARY OF THE INVENTION

[0009] The present invention relates to a process for converting wax to high quality lube basestocks by contacting the wax with a unidimensional molecular sieve catalyst with a near circular pore structure having an average diameter of 0.50 nm to 0.65 nm wherein the difference between the maximum diameter and the minimum is ≤ 0.05 nm (e.g., ZSM-48) followed by a second molecular sieve catalyst (e.g., Zeolite Beta). Both catalysts comprise one or more Group VIII metals (i.e., Fe, Ru, Os, Co, Rh, Ir, Pd, Pt, Ni).

BRIEF DESCRIPTION OF THE FIGURES

[0010] Figure 1 is a plot of lube yield versus lube pour point for isomerization of C80 wax over Pt/ZSM-48 followed by Pt/Beta, and stand-alone Pt/ZSM-48 catalyst systems.

[0011] Figure 2 is a plot of lube viscosity versus lube pour point for isomerization of C80 wax over Pt/ZSM-48 followed by Pt/Beta, and stand-alone Pt/ZSM-48 catalyst systems.

[0012] Figure 3 is a plot of viscosity index (VI) versus lube pour point for isomerization of C80 wax over Pt/ZSM-48 followed by Pt/Beta, and stand-alone Pt/ZSM-48 catalyst systems.

[0013] Figure 4 is a plot of light gas yield versus lube pour point for isomerization of C80 wax over Pt/ZSM-48 followed by Pt/Beta, and stand-alone Pt/ZSM-48 catalyst systems.

[0014] Figure 5 is a plot of naphtha yield versus lube pour point for isomerization of C80 wax over Pt/ZSM-48 followed by Pt/Beta, and stand-alone Pt/ZSM-48 catalyst systems.

[0015] Figure 6 is a plot of diesel yield versus lube pour point for isomerization of C80 wax over Pt/ZSM-48 followed by Pt/Beta, and stand-alone Pt/ZSM-48 catalyst systems.

DETAILED DESCRIPTION

[0016] The invention provides high isomerization and dewaxing selectivity of a wax over an unidimensional catalyst with a near circular pore structure having an average pore diameter of 0.50-0.65 nm (5.0-6.5 angstroms) wherein the maximum diameter - minimum diameter \leq 0.05 nm (0.5 angstroms), followed by a molecular sieve catalyst to form a lubricant. Group VIII metals on the two catalysts are preferred and platinum is the most preferred.

[0017] The invention improves lube basestock products and their properties (e.g., pour point, cloud point). This method effectively reduces average lube molecular weight and potentially reduces lube product cloud point without sacrificing lube yield. This process allows improved use of the heavy end of lubes and is especially suited for waxes with 1,000°F+ fractions and preferably 1,100°F+ fractions. These fractions may comprise the higher molecular weight or boiling point tail of the feeds. It would be difficult to create enough

branches with minimal cracking for very large molecular weight feeds if only one of the above catalysts was used. This invention is preferably used for processing heavy lube or lubes with a heavy component (e.g., with >5 wt% heavy raffinate) where the Beta catalyst selectively cracks the heavy end. This invention can give a lighter lube with yields similar to those obtained over ZSM-48 alone.

[0018] Preferably, wax feed is first passed over a ZSM-48 catalyst. The resulting intermediate product is then passed over a single Zeolite Beta catalyst to form the final lube. These first and second stages can be separated or preferably are integrated process steps (e.g., cascaded).

[0019] The unidimensional molecular sieve catalyst with near-circular pore structures does most of the dewaxing. The pores are smaller than in large pore molecular sieves thereby excluding bulkier (e.g., highly branched) molecules. Unidimensional means that the pores are essentially parallel to each other.

[0020] The pores of the catalyst have an average diameter of 0.50 nm to 0.65 nm wherein the difference between a minimum diameter and a maximum diameter is \leq 0.05 nm. The pores may not always have a perfect circular or elliptical cross-section. The minimum diameter and maximum diameter are generally only measurements of an ellipse of a cross-sectional area equal to the cross-sectional area of an average pore. The pores can alternatively be defined by finding the center of the pore cross-section and using half of the minimum diameter and half of the maximum diameter to sweep an average cross-sectional pore shape from the center.

[0021] The preferred unidimensional molecular sieve catalyst is an intermediate pore molecular sieve catalyst of which the preferred version is ZSM-48. U.S. Patent 5,075,269 describes the procedures for making ZSM-48 and is incorporated by reference herein. ZSM-48 is roughly 65% zeolite crystal and 35% alumina. Of the crystals, at least 90%, preferably at least 95%, and most preferably 98-99% are ideal crystals. The ZSM-48 is preferably in the protonated form though some sodium is acceptable. ZSM-48 is more robust than other catalysts with similar functions and helps to protect the second catalyst (e.g., Zeolite Beta).

[0022] In the first stage of the process, the unidimensional intermediate pore molecular sieve catalyst (e.g., Pt/ZSM-48) is preferably kept at 500-800°F (260-427°C), more preferably at 600-700°F (316-371°C), and most preferably at 630-660°F (332-349°C). ZSM-48 catalysts used in the invention preferably have an Alpha value of about 10 to about 50 prior to the Group VIII metal loading.

[0023] Zeolite Beta catalysts are 12 ring acidic silica/alumina zeolites with or without boron (replacing some of the aluminum atoms). Zeolite Y (USY), though less preferred than Beta, is also contemplated in the scope of the invention. Pre-sulfided Zeolite Beta is preferred when some residual sulfur in the product is acceptable.

[0024] Zeolite Betas used in the invention preferably have an Alpha value below 15, more preferably below 10, at least prior to metal loading. Alpha is an acidity metric that is an approximate indication of the catalytic cracking activity of the catalyst compared to a standard catalyst. Alpha is a relative rate constant (rate of normal hexane conversion per volume of catalyst per unit

time). Alpha is based on the activity of the highly active silica-alumina cracking catalyst taken as an Alpha of 1 in U.S. Patent 3,354,078 (incorporated by reference) and measured at 538°C as described in the Journal of Catalysis, vol. 4, p. 527 (1965); vol. 6, p. 278 (1966); and vol. 61, p. 395 (1980). Feeds with minimal nitrogen content will require low Alpha value of this catalyst. In comparison, catalysts with high Alpha values are used for less selective cracking. Alpha values may be reduced by steaming.

[0025] The Beta catalyst (e.g., Pt/Beta), when contacting the intermediate product, is most preferably kept at temperatures of 400-700°F (204-371°C), more preferably at 500-650°F (260-343°C), and most preferably at 520-580°F (271-304°C).

[0026] The temperature of each catalyst is preferably controlled independently. Temperature choice partly depends on the feed liquid hourly space velocity of which 0.1-20 h⁻¹ is preferred, 0.5-5 h⁻¹ is more preferred, and 0.5-2 h⁻¹ is most preferred.

[0027] The contact time for both catalysts is preferably similar to each other. It is understood that the space velocity can be different. The pressure for both catalysts is preferably similar to each other. Hydrogen co-feed flow rate is 100-10,000 scf/bbl (17.8-1,780 n.L.L⁻¹), more preferably 1,000-6,000 scf/bbl (178-1,068 n.L.L⁻¹), and most preferably 1,500-3,000 scf/bbl (267-534 n.L.L⁻¹).

[0028] Each catalyst comprises 0.01-5 wt% of at least one Group VIII metal (i.e., Fe, Ru, Os, Co, Rh, Ir, Pd, Pt, Ni). Platinum and palladium are most preferred. Platinum or palladium blended with each other or other group VIII

metals follow in preference. Nickel may also be blended with group VIII precious metals and is included in the scope of the invention whenever group VIII blends, alloys, or mixtures are mentioned. Platinum is the most preferred metal. Preferred metal loading on both catalysts are 0.1-1 wt% with approximately 0.6 wt% most preferred.

[0029] The feed preferably is a wax with a melting point over 50°C, less than 7,000 ppm sulfur, and less than 50 ppm nitrogen. The nitrogen is more preferably less than 10 ppm nitrogen if hydrogen pressure is below 500 psig (34 atm). For example, a heavy raffinate can be blended with a Fischer-Tropsch wax or similar clean waxy feed (e.g., to lower sulfur and/or nitrogen levels).

[0030] The feed is converted by the first catalyst to form an intermediate product which is then preferably passed directly from the first catalyst to the second catalyst. In a preferred embodiment of the invention, a cascaded two-bed catalyst system consisting of a first bed catalyst followed by a second bed catalyst allows a highly selective process for wax isomerization and lube hydrodewaxing with minimal gas formation. In cascading, the intermediate product preferably directly passes from the first bed to the second bed without inter-stage removal of light products. Optionally, light byproducts (e.g., methane, ethane) can be removed between the first and second catalysts.

[0031] More branching in feeds facilitates the present invention and improves final lube yield. U.S. Patent 6,090,989 describes typical branching indices and is incorporated by reference. The feed is preferably mixed with hydrogen and preheated before contacting it with the first catalyst. Preferably,

at least 95% of the wax is in liquid form before contacting it with the first catalyst.

[0032] The preferred measurements, as taught by the specification, are described in this paragraph. Where there are two values, the value in parenthesis is approximate metric conversion of the first value. The weight percent of paraffins may be measured by high-resolution $^1\text{H-NMR}$, for example, by the method described in ASTM standard D5292, in combination with GC-MS. This approach may also be used to determine the weight percentage of unsaturates, alcohols, oxygenates, and other organic components. The iso- to normal-paraffin ratio may be measured by performing gas chromatography (GC) or GC-MS in combination with $^{13}\text{C-NMR}$. Sulfur may be measured by XRF (X-Ray Fluorescence), as described, for example, in ASTM standard D2622. Nitrogen may be measured by syringe/inlet oxidative combustion with chemiluminescence detection, for example, by the method described in ASTM standard D4629. Aromatics may be measured as described below. As taught by the specification, olefins may be measured by using a Bromine index determined by coulometric analysis, for example, by using ASTM standard D2710. The weight percent of total oxygen may be measured by neutron activation in combination with high-resolution $^1\text{H-NMR}$. If necessary, the total oxygen content may be placed on a water-free basis by measuring water content. For samples having a water content known to be less than about 200 ppm by weight, one may use known derivitization methods (e.g., by using calcium carbide to form acetylene) followed by GC-MS. For samples having a water content known to be greater than about 200 ppm by weight, one may use the Karl-Fischer method, for example, by the method described in ASTM standard D4928. The total alcohol content may be determined by high-resolution $^1\text{H-NMR}$, and the percentage present primarily

as C₁₂-C₂₄ primary alcohols may be determined by GC-MS. Cetane number may be determined by using, for example, ASTM standard D613. The level of aromatics may be determined by using high-resolution ¹H-NMR, for example, by using ASTM standard D5292. Dioxygenates are measured by using infrared (IR) absorbance spectroscopy. Branching characteristics of iso-paraffins may be measured by a combination of high-resolution ¹³C-NMR and GC with high-resolution MS.

EXPERIMENTAL

[0033] A cascaded two-bed catalyst system consisting of a first stage Pt/ZSM-48 catalyst immediately followed by a second stage of Pt/Beta catalyst is shown to be highly active and selective for hydroisomerization and dewaxing of waxes with high molecular weight components.

[0034] Example operating conditions, material balance data, lube yields and properties are summarized in Table 1. TBP x% indicates temperature below which x wt% of hydrocarbon samples boils. Time on stream (TOS) is the time during which the feed contacts the catalyst. IBP is initial boiling point. TBP is terminal boiling point. The best S.I. equivalent of standard cubic feed of hydrogen per barrel of feed (SCF/bbl) is normal liters of hydrogen gas per liter of feed (n.l.l⁻¹. or n.L.L⁻¹ or n.L (gas) / L (feed)). LHSV is defined as liquid hourly space velocity. WHSV is defined as weight hourly space velocity.

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TABLE 1
Hydroisomerization of C80 Wax Catalyzed by a Cascaded Pt/ZSM-48 Followed by Pt/Beta
(1.0 h⁻¹ LHSV for Each Catalyst)

Run Number, 401-		3-3	3-11	3-16	3-20	3-22	3-24
Time on Stream, Days		3.6	15.1	21.6	26.5	28.6	31.1
ZSM-48 Temperature, °F		660	660	640	655	645	640
(ZSM-48 Temperature, °C)	approximate	349	349	338	346	341	338
Beta Temperature, °F		560	560	540	560	560	560
(Beta Temperature, °C)	approximate	293	293	282	293	293	293
Pressure, psig		1000	1000	1000	1000	1000	1000
(Pressure, atm)	approximate	68	68	68	68	68	68
H ₂ Cofeeding Rate, scf/bbl		5786	6150	5575	5528	5607	5619
(H ₂ Cofeeding Rate, n.L.L. ⁻¹)	approximate	1030	1095	992	984	5607	1000
700°F+(371°C) Conversion, wt%		83.5	79.4	34.6	60.7	47.7	40.4
H ₂ Consumption, scf/bbl		499	516	205	377	270	225
(H ₂ Consumption, n.L.L. ⁻¹)	approximate	89	92	36	67	48	40
Product Yield, wt% on Feed							
C ₁ -C ₄ Gas		4.0	6.2	3.2	5.7	3.4	2.8
C ₅ -330°F (C ₅ -166°C) Naphtha		33.4	31.2	9.6	18.2	13.2	11.4
330-700°F (166-371°C) Diesel		47.0	42.9	22.1	37.5	31.6	26.6
700°F+(371°C+) Lube		16.5	20.6	65.4	39.3	52.3	59.6
Total Hydrocarbon		100.9	101.0	100.4	100.7	100.5	100.4

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TABLE 1 (continued)

Run Number, 401-		3-3	3-11	3-16	3-20	3-22	3-24
700°F+(371°C+) Lube Properties	Feed						
KV @ 40°C, cSt		34.7	24.8	34.0	28.1	28.8	28.3
KV @ 100°C, cSt		9.4	6.31	5.06	6.91	5.77	6.00
Viscosity Index		133.5	136.0	168.7	153.4	159.8	165.2
Pour Point, °C		82	-60	-54	0	-33	-21
Cloud Point, °C			-60	-54	13	0	-10
TBP 5%, °F		754	702	783	723	719	716
(TBP 5%, °C)	approximate	401	372	417	384	382	380
TBP 50%, °F		875	840	922	877	879	895
(TBP 50%, °C)	approximate	468	449	494	469	471	479
TBP 95%, °F		1004	1006	1062	1030	1019	1028
(TBP 95%, °C)	approximate	540	541	572	554	548	553
MB Closure, wt%		97.6	95.6	98.2	98.5	98.0	98.1

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[0035] To obtain desirable wax isomerization results, a mild (e.g., 500-630°F (260-332°C)) Pt/Beta temperature should be employed during lube hydroprocessing. The mild Pt/Beta temperature should be employed with varying Pt/ZSM-48 temperature to achieve a target lube pour point. To achieve maximal lube yield, low operating pressure (< 2,000 psi (272 atm) hydrogen pressure) should be used.

[0036] Stand-alone Pt/ZSM-48 was also evaluated for isomerizing and dewaxing C80 wax to 700°F+ (371°C+) lube basestocks (Tables 2). Comparison of lube yields for the two catalyst systems is illustrated in Figure 1. Figure 1 shows that the cascaded Pt/ZSM-48 followed by Pt/Beta gave essentially identical lube yields compared to Pt/ZSM-48 alone. The addition of Pt/Beta had minimal effects on the range of Pt/ZSM-48 operating temperature (Tables 1 and 2).

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TABLE 2**Hydroisomerization of C80 Wax Catalyzed by Pt/ZSM-48**

Run Number, 401-		3-27	3-28	3-29	3-30	3-31
Time on Stream, Days		35.6	37.0	38.0	39.0	40.9
Temperature, °F		665	660	655	650	645
Temperature, °C	approximate	352	349	352	343	341
Pressure, psig		1000	1000	1000	1000	1000
(Pressure, atm)	approximate	68	68	68	68	68
LHSV, hr ⁻¹		1.0	1.0	1.0	1.0	1.0
WHSV, hr ⁻¹		1.4	1.5	1.5	1.4	1.4
H ₂ Cofeeding Rate, scf/bbl		5656	5643	5603	5674	5657
(H ₂ Cofeeding Rate, n.L.L. ⁻¹)	approximate	1007	1004	997	1010	1007
700°F+ (371°C+) Conversion, wt%		78.0	70.6	60.0	49.9	44.2
H ₂ Consumption, scf/bbl		544	473	377	306	261
(H ₂ Consumption, n.L.L. ⁻¹)	approximate	97	84	67	54	46
Product Yield, wt% on Feed						
C ₁ -C ₄ Gas		8.3	6.8	5.4	4.4	3.5
C ₅ -330°F (C ₅ -166°C) Naphtha		30.0	26.1	19.6	15.6	13.7
330-700°F (166-371°C) Diesel		40.8	38.6	35.7	30.4	27.5
700°F+ (371°C+) Lube		22.0	29.4	40.0	50.1	55.8
Total Hydrocarbon		101.0	100.9	100.7	100.6	100.5

TABLE 2 (continued)

Run Number, 401-		3-27	3-28	3-29	3-30	3-31
700°F+ (371°C+) Lube Properties						
KV @ 40°C, cSt	Feed	14.8	34.8	31.2	32.9	34.0
KV @ 100°C, cSt	9.4	3.65	6.59	6.29	6.66	6.90
Viscosity Index	135.5	147.4	156.9	163.8	168.6	
Pour Point, °C	82	-54	-48	-33	-24	-12
TBP 5%, °F	570	778	753	766	770	
(TBP 5%, °C)	299	414	400	407	410	
TBP 50%, °F	783	899	906	918	918	
(TBP 50%, °C)	417	482	485	492	492	
TBP 95%, °F	998	997	1007	1014	1057	
(TBP 95%, °C)	537	536	542	546	569	
MB Closure, wt%	99.6	98.8	98.8	97.9	97.1	

[0037] The viscosity and viscosity index of the nominal 700°F+ (371°C+) C80 wax isomerates vs. hydroprocessing severity are plotted in Figures 2 and 3, respectively. The two sets of data compared in the two diagrams correspond to the wax isomerates prepared using Pt/ZSM-48 followed by Pt/Beta, and stand-alone Pt/ZSM-48.

[0038] As shown in Figure 2, the Pt/ZSM-48-Pt/Beta isomerates had significantly lower viscosities presumably due to the relatively high cracking activity of Pt/Beta catalyst towards multi-branched isoparaffins. Thus, this dual catalysts system provides an effective method for reducing average molecular weight of a wax feed to produce lower viscosity lube stocks without sacrificing lube yield during wax hydroisomerization process.

[0039] Figure 3 shows high viscosity indices observed for the Pt/ZSM-48-Pt/Beta wax isomerates, although they are slightly lower than those of Pt/ZSM-48 isomerates. For products of the invention, a viscosity index of at least 150 at a -20°C lube pour point and a viscosity index of at least 130 at a pour point of no more than -50°C is preferred.

[0040] The spread between the lube cloud and pour points for Pt/ZSM-48-Pt/Beta is mostly less than 15°C (Table 1). In general, the spread between the lube cloud and pour points narrows with decreasing pour point.

[0041] The overall light byproduct selectivity for the two catalyst systems is comparable (Figures 4-6). As expected, the yields of gases, naphtha, and diesel increase for both systems with increasing process severity (decreasing lube pour point) that promotes hydrocracking.

[0042] The following examples will serve to illustrate the invention.

Example 1

[0043] *Feedstock.* The hydrotreated SASOLTM PARAFLINTTM C80 wax (C80) feed was obtained from Moore and Munger, Inc., (Shelton, CT) and used as received without additional pretreatment. The C80 wax was a mixture of predominantly linear paraffins with very low content of olefins and oxygenates. SASOLTM has been marketing three commercial grades of waxes: PARAFLINTTM H1, a 700°F+ (371°C+) full range wax; PARAFLINTTM C80 and C105, 700-1100°F (371-593°C) and 1100°F+ (593°C+) distillate fractions, respectively. The molecular weight distribution (in terms of boiling point) of the waxes is illustrated briefly in Table 3.

TABLE 3
Molecular Weight Distribution of Waxes in Examples

Wax Feed	H1	C80	C105
Pour Point, °C	99	82	106
IBP-700°F (<C ₂₄), wt%	0	3	0
700-1100°F (C ₂₄ -C ₆₀), wt%	44	89	20
1100°F+ (>C ₆₀), wt%	56	8	80

Example 2

[0044] *Preparation of Pt/Beta Catalyst.* Pt/Beta catalyst was prepared by extruding a water-containing mull mix or paste containing 65 parts of Zeolite Beta with 35 parts of alumina (dry basis). After drying, the Zeolite Beta containing catalyst was calcined under nitrogen at 900°F (482°C) and exchanged at ambient temperature with a sufficient quantity of ammonium

nitrate to remove residual sodium in the zeolite channels. The extrudate was then washed with de-ionized water and calcined in air at 1000°F (538°C). After air calcination, the 65% Zeolite Beta/35% Alumina extrudate was steamed at 1020°F (549°C) to reduce the Alpha value of the calcined catalyst to less than 10. The steamed, 65% low acidity Beta/35% Alumina catalyst was ion exchanged with a tetraammine platinum chloride solution under ion exchange conditions to uniformly produce a catalyst containing 0.6% Pt. After washing with de-ionized water to remove residual chlorides, the catalyst was dried at 250°F (121°C) followed by final air calcination at 680°F (360°C).

Example 3

[0045] Preparation of Pt/ZSM-48 Catalyst. Pt/ZSM-48 catalyst was prepared by extruding a water-containing mull mix or paste containing 65 parts of ZSM-48 with 35 parts of alumina (dry basis). After drying, the ZSM-48 containing catalyst was calcined under nitrogen at 900°F (482°C) and exchanged at ambient temperature with a sufficient quantity of ammonium nitrate to remove residual sodium in the zeolite channels. The extrudate was then washed with deionized water and calcined in air at 1000°F (538°C). After air calcination, the 65% ZSM-48 / 35% Alumina catalyst was impregnated with a tetraammine platinum nitrate solution under incipient wetness conditions to uniformly produce a catalyst containing 0.6% Pt. Finally, the catalyst was dried at 250°F (121°C) followed by air calcination at 680°F (360°C).

Example 4

[0046] *Wax Hydroprocessing.* The wax hydroisomerization experiments were performed using a micro-unit equipped with two three-zone furnaces and two down-flow trickle-bed tubular reactors (1/2" ID) in cascade (with option to bypass the second reactor). The unit was carefully heat-traced to avoid freezing of the high melting point C80 wax. To reduce feed bypassing and lower zeolite pore diffusion resistance, the catalysts extrudates were crushed and sized to 60-80 mesh. The reactors 1 and 2 were then loaded with 15 cc of the 60-80 mesh Pt/ZSM-48 catalyst and the 60-80 mesh Pt/Beta catalyst, respectively. 5 cc of 80-120 mesh sand was also added to both catalyst beds during catalyst loading to fill the void spaces. After pressure testing of the unit, the catalysts were dried and reduced at 400°F (204°C) for one hour under 1 atmosphere (atm), 255 cc/min hydrogen flow. At the end of this period, the flow of pure hydrogen was stopped and flow of H₂S (2% in hydrogen) was initiated at 100 cc/min. After H₂S breakthrough, the reactors 1 and 2 were gradually heated to 700°F (371°C) and maintained at 700°F (371°C) for 1 h (hour). After the completion of catalyst pre-sulfiding, the gas flow was switched back to pure hydrogen at 255 cc/min rate, and the two reactors were cooled down.

[0047] Hydroisomerization of the C80 wax over a cascaded Pt/ZSM-48 followed by Pt/Beta was conducted at 1.0 h⁻¹ LHSV for each catalyst and 1000 psig (68 atm) with 5500 scf/bbl (979 n.L.L⁻¹) hydrogen circulation rate. The wax isomerization experiments were started first by saturating the catalyst beds with the feed at 400°F (204°C) then heating the reactors to the initial operating temperatures. Material balances were carried out overnight for 16-24 h. Reactor temperatures were then gradually changed to vary pour point.

[0048] Performance of stand-alone Pt/ZSM-48 was evaluated by cooling and bypassing the Pt/Beta catalyst in the second reactor. The experiments were conducted under identical process conditions (1.0 LHSV, 1000 psig (68 atm), 5500 scf/bbl (979 n.L.L⁻¹) H₂) and according to similar procedures used for testing the cascade Pt/ZSM-48 and Pt/Beta combination.

Example 5

[0049] *Product Separation and Analysis.* Off-gas samples were analyzed by GC using a 60m DB-1 (0.25 mm ID) capillary column with FID detection. Total liquid products (TLP's) were weighed and analyzed by simulated distillation (Simdis, such as D2887) using high temperature GC. TLP's were distilled into IBP-330°F (IBP-166°C) naphtha, 330-700°F (166-371°C) distillate, and 700°F+ (371°C+) lube fractions. The 700°F+ (371°C+) lube fractions were again analyzed by Simdis to ensure accuracy of the actual distillation operations. The pour point and cloud point of 700°F+ (371°C+) lubes were measured by D97 and D2500 methods, and their viscosities were determined at both 40°C and 100°C according to D445-3 and D445-5 methods, respectively.